

⑯



Europäisches Patentamt

European Patent Office

Office européen des brevets

⑯ Publication number:

0 320 552

A1

⑯

EUROPEAN PATENT APPLICATION

⑯ Application number: 87311191.8

⑯ Int. Cl. 4: **B05D 7/16, B05D 7/26**

⑯ Date of filing: 18.12.87

⑯ Date of publication of application:
21.06.89 Bulletin 89/25

⑯ Designated Contracting States:
BE DE ES FR GB IT

⑯ Applicant: **E.I. DU PONT DE NEMOURS AND COMPANY**
1007 Market Street
Wilmington Delaware 19898(US)

⑯ Inventor: **Chang, David Chi Kung**
5554 Lahser Road
Birmingham Michigan 48010(US)

⑯ Representative: **Blake, John Henry Francis et al**
BROOKES AND MARTIN High Holborn House
52/54 High Holborn
London WC1V 6SE(GB)

⑯ A process for improving the appearance of a multilayer finish.

⑯ An improved process for applying a multilayer finish on a primer coated substrate by using a guide coating composition that is applied over the primer of the substrate and then a water borne base coating composition containing metallic pigments and a clear coating composition are applied; before the base coating composition is applied, the guide coating composition is dried at ambient temperatures or at elevated temperatures but not fully cured or crosslinked; the guide coating contains a polymeric latex in an aqueous carrier, preferably an acrylic latex, and optionally, a crosslinking agent and metallic flake pigments; the process improves the appearance, particularly head-on-brightness of water borne basecoat/clear coat finishes in which the base coat contains metallic flake pigments.

EP 0 320 552 A1

Xerox Copy Centre

A PROCESS FOR IMPROVING THE APPEARANCE OF A MULTILAYER FINISH

BACKGROUND OF THE INVENTION

5 This invention is related to an improved process for forming multilayer finishes on automobiles and trucks. In particular, the process forms finishes that have an improved appearance.

Multilayer finishes, in particular, clear coat/base coat finishes are well known in the art, as shown, for example, in Benefiel et al US 3,639,147, issued February 1, 1972. Generally, solvent based coating compositions have been used to form these multilayer finishes. To comply with current air quality 10 standards, water borne base coat finishes in combination with either solvent based or water based clear coat finishes are being tested for use. However, it would be desirable to improve the appearance of water borne base coat finishes containing metallic flake pigments. In particular, improvements in head-on-brightness would be desired. This invention provides an improved process and a composition that can be applied by this process to form a clear coat/base coat finish with water borne base coating compositions 15 containing metallic flake pigments that have a high quality appearance that is acceptable to the automotive industry.

SUMMARY OF THE INVENTION

20

This invention is directed to an improved process for applying a multilayer finish to a substrate that has a primer layer adhered thereto and a layer of a water borne base coating composition containing metallic flake pigments and a layer of a clear top coating composition; the improvement that is used with this 25 process comprises the following:

applying a layer of a waterborne guide coating composition containing an aqueous carrier and a polymeric binder to the primer layer before application of the water borne base coating composition and drying the guide coating at ambient temperatures or at an elevated temperature without crosslinking or curing the guide coating and then applying the waterborne base coating composition to the guide coating and clear 30 top coating composition to the base coating and baking at elevated temperatures to fully cure the resulting multilayer finish; whereby the resulting multilayer finish on the substrate comprising the guide coating, base coating and clear topcoating has an improved appearance.

35

DETAILED DESCRIPTION OF THE INVENTION

The improved process of this invention is used to improved the appearance of a water borne base coating composition/clear coating composition used for automobiles and trucks. In this process, a guide 40 coating is applied over the primer coating of the substrate and then dried but not cured. The guide coat can be dried at ambient temperatures e.g. 20-25° C or baked at elevated temperatures up to 180° C for a short time or exposed to infrared radiation for rapid drying taking care not to crosslink the guide coat. Then the water borne base coating is applied over the guide coating and then the clear coating composition is applied over the base coating and the resulting coated substrate is baked at an elevated temperature to 45 form a fully cured multilayer finish. The resulting base coat/clear coat finish has an improved appearance, particularly for head-on-brightness, in comparison to clear coat/color coat finishes that do not use a guiding coating composition.

The guide coating composition is an aqueous based composition containing about 10-75% by weight of a film forming binder and preferably, a crosslinking agent and pigments. Typically, an aqueous latex is used 50 as the film forming binder and preferably, an acrylic latex is used. An alkylated melamine formaldehyde crosslinking agent can be used in the composition. Any of the conventional pigments used for coating compositions or primers can be used in the composition.

Usually, the binder of the guide coating composition is an acrylic latex but other water dispersible or water soluble polymers can be used. The latex is prepared by a conventional emulsion polymerization process in which monomers, a polymerization catalyst such as ammonium persulfate, water, surfactants and

usually a neutralizing agent such as ammonia or an amine such as amino ethyl propanol are utilized. The polymerization is carried out under conventional temperatures of about 50-90° C for about 1-5 hours to form a latex.

5 The monomers used for the latex are alkyl methacrylates, alkyl acrylates, hydroxy alkyl acrylates and methacrylates and an ethylenically unsaturated mono or di carboxylic acids. Difunctional acrylates or methacrylates also can be used.

Typical alkyl methacrylates are methyl methacrylate, ethyl methacrylate, propyl methacrylate, butyl methacrylate, hexyl methacrylate, octyl methacrylate, nonyl methacrylate, lauryl methacrylate and the like.

10 Typical alkyl acrylates are methyl acrylate, ethyl acrylate, propyl acrylate, butyl acrylate, hexyl acrylate, octyl acrylate, nonyl acrylate, lauryl acrylate and the like.

Typical hydroxy alkyl acrylates and hydroxy alkyl methacrylates that can be used are hydroxy ethyl methacrylate, hydroxy propyl methacrylate, hydroxy butyl methacrylate, hydroxy ethyl acrylate, hydroxy propyl acrylate, hydroxy butyl acrylate and the like.

15 Typical polymerizable mono or di carboxylic acids that can be used are methacrylic acid, acrylic acid, itaconic acid, maleic acid and the like.

Difunctional monomers that can be used are allyl methacrylate, allyl acrylate and other diacrylates. One preferred latex polymer contains 10-20% by weight methyl methacrylate, 70-80% by weight butyl acrylate, 2-9% by weight hydroxy ethyl acrylate, and 1-5% by weight methacrylic acid.

20 The latex itself without a crosslinking agent can be used as a guide coating composition. This has an advantage that the latex does not crosslink on drying or baking at an elevated temperature.

Generally, the guide coating contains about 5-40% by weight, based on the weight of the binder, of the crosslinking agent. One preferred composition contains about 5-30% by weight of the crosslinking agent and 70-95% of the latex polymer.

25 Typical monomeric and polymeric alkylated melamine formaldehyde crosslinking agents that can be used are partially or highly methylated melamine formaldehyde resins, such as "Cymel" 300 resins, mixed ether and butylated melamine formaldehyde resins or other partially or fully alkylated melamine formaldehyde resins having 1-6 carbons in the alkyl group. One preferred resin is "Cymel" 325 which is a methylated melamine formaldehyde resin that has a methoxymethyl-imino functionality. Another preferred resin is "Cymel" 303 which is a highly methylated melamine formaldehyde resin containing reactive alkoxy groups.

30 The guide coating composition can be used as an unpigmented clear but preferably contains pigments in a pigment to binder weight ratio of about 0.5/100 to 300/100. Generally, the pigments provide additional hiding and are usually the same as the pigments of the base coating composition. Typical pigments that can be used are titanium dioxide, other metallic oxides such as zinc oxide and iron oxide, carbon black, the organic pigments and dyes, metallic flake pigments such as aluminum flake, filler pigments, silica, and the like. Preferably, the composition contains metallic flake pigments such as aluminum flake. The pigments are formulated into a millbase using conventional procedures and then the mill base is blending with the latex and the crosslinking agent to form the guide coating composition.

35 The substrates over which the guide coat is applied are those conventionally used for automotive and truck bodies such as cold roll steel, phosphatized steel, polyester reinforced fiber glass, reaction injection molded urethanes, crystalline amorphous polyamides and the like. Typically, these substrates are coated with alkyd resin, epoxy resin or polyester primers.

40 After the primer is applied to the substrate and cured, the guide coating composition is applied. Any of the conventional methods of application can be used such as spraying or electrostatic spraying. The coating is applied to provide a dry film thickness of about 0.1 to 1.0 mils. The coating is then dried but not fully cured or crosslinked. The base coating composition is applied by spraying or electrostatic spraying to provide a dry film of about 0.2-1.5 mils thick. A clear coating composition is applied by spraying or electrostatic spraying to a dry film thickness of about 0.5-6.0 mils. The resulting multilayer finish then is baked at about 120-175° C for about 15 minutes - 1 hour to form a glossy finish having excellent head-on-brightness and a good appearance.

45 The base coating composition can be any of the composition that are conventionally used such as acrylics or polyesters containing crosslinking agents. These compositions can be solvent or water based solutions or dispersions. The process of this invention is particularly useful for waterborne base coatings containing metallic flake pigments and provides a finish with improved head-on-brightness. However, improvements are noted also with the use of solvent based base coat finishes.

50 The clear coating composition can be a solvent or water based composition of an acrylic or polyester polymer and containing a crosslinking agent or other type of composition as are conventionally used for clear coating compositions.

55 The guide coating composition also can be used with two component base coat/clear coat compositions

The guide coating composition

that do not require baking for curing such as two component acrylic urethanes, acrylic esters, acrylic alkyds, epoxy esters and the like. These two component compositions are used to refinish cars and trucks. Application of the guide coating composition is the same as above and then the two component base coating composition and clear coating composition are applied and cured at ambient temperatures or force dried at elevated temperatures.

The following examples illustrate the invention. All parts and percentages are on a weight bases unless otherwise indicated.

19

EXAMPLE

A latex was prepared by charging the following ingredients into a polymerization vessel equipped with a stirrer and a heating source:

15

	Portion 1	Parts By Weight
20	Deionized water Alkyl phenol ethylene oxide anionic surfactant	2535.0 16.4
	Portion 2	
25	Anionic surfactant (described above) Methyl methacrylate monomer Butyl methacrylate monomer Deionized water	23.4 369.0 1622.4 2335.0
	Portion 3	
30	Deionized water Ammonium persulfate	225.0 7.0
	Portion 4	
35	Deionized water Anionic surfactant (described above) Methacrylic acid Hydroxy ethyl acrylate monomer Butyl acrylate monomer	1097.0 11.7 81.7 116.8 385.3
	Portion 5	
40	Deionized water Ammonium persulfate	85.0 2.3
	Portion 6	
45	Deionized water Amino ethyl propanol "Cosan" 145 - latex preservative of an oxazolidine derivative made by Cosan Chemical Corp. Total	451.0 60.7 10.0 9434.6

50 Portion 1 is charged into the polymerization vessel and heated to about 83°C. Portion 2 is premixed. About 10% of Portion 2 is added and the resulting reaction mixture is held at about 80°C. About 10% of Portion 3 is added and the reaction mixture is held at the above temperature. The remainder of Portion 2 is added over a 60 minute period along the about 72% of Portion 3 while controlling the reaction mixture at about 85°C. The remainder of Portion 3 along with Portion 4 is added over a 30 minute period while controlling the reaction mixture at 85°C. Portion 5 is added and the reaction mixture is held at about 85°C for 60 minutes. Portion 6 is added over a 30 minute period and held at the above temperature for about 1 hour. The resulting latex is cooled to room temperature and filtered.

The latex has a polymer solids content of 27.7% and a pH of 7.2. The polymer is comprised of 14.3% methyl methacrylate, 78.0% butyl acrylate, 3.2% methacrylic acid, and 4.5% hydroxy ethyl acrylate.

5 A mill base was prepared as follows:

5			Parts By Weight
			Ethylene glycol monobutyl ether
		Polyoxyethylene ester of mixed fatty acids and resins	4.87
		Aluminun flake paste - 65% solids aluminuN flake in mineral spirits	40.00
		Total	100.00

10

The above constituents are charged into a mixing vessel and thoroughly mixed together. The following guide coating compositions A-C were formulated:

15	COMPOSITION	Parts By Weight		
		A	B	C
20	Latex (prepared above)	1557.0	1557.0	1557.0
25	Deionized water	488.0	1029.0	1029.0
30	Melamine formaldehyde resin solution (80% solids in isobutanol of a highly methylated melamine formaldehyde resin having a low methylol content and containing alkoxy imino groups)	104.0	104.0	-----
35	Mill base (prepared above)	331.0	----	----
40	Deionized water	541.0	----	----
45	Dimethyl ethanol amine "Acrysol" ASE 60 (Acrylic latex thickener)	23.4	23.4	23.4
	Total	28.0	28.0	28.0
		3072.4	2741.4	2637.4

50 Each of the above Guide Coating Compositions A-C were sprayed onto separate phosphatized steel panels coated with an epoxy/polyester electrodeposition primer. Each composition was sprayed onto four separate panels. One panel was dried at ambient temperature of about 25 °C for about 30 minutes; the second panel was baked for about 2 minutes at about 104 °C which was sufficient time to dry the coating but not cure the coating; the third panel was baked for about 10 minutes at about 121 °C which was sufficient time to cure the coating and the fourth panel was baked for about 30 minutes at 121 °C which was sufficient to cure the coating. In each case the dried Guide Coating was about 0.3 mils thick when dried.

55 A base coating was then applied by spraying a layer of above Composition A to each of the above coated panels and the base coating was dried at ambient temperatures for about 10 minutes to form a dried coating about 0.5 mils thick. Then a clear topcoating was spray applied and the coated panels were baked

for about 30 minutes at about 120° C to form a clear coat/base coat finish.

The above clear coating composition has a solids content of about 68% in a 50/50 solvent mixture of methyl amyl ketone and aromatic hydrocarbon wherein the polymer comprises styrene/butyl methacrylate/butyl acrylate/hydroxy ethyl acrylate/acrylic acid in a ratio of about 14.9/28/30/25/2.1.

5 A control for each of the guide coats A - C of phosphatized primed steel panel was prepared as above i.e. the panel was coated with the base coat and the clear coat as above, except a guide coat was omitted. Each panel was baked under the same conditions. These panels were used as the controls and represents prior art method of preparing a clear coat/base coat finish.

10 The head on brightness was measured using the spectrophotometer described in Lee et al. U.S. Patent 4,412,744 issued November 1, 1983 for each of the above prepared panels and the results are as follows:

15

20

25

30

35

40

45

50

55

GUIDE COATING	DRY AMBIENT TEMP/FAKE (NO GUIDE COATING) 30° X AMB. TEMP.	HEAD-ON-BRIGHTNESS	NOTE
CONTROL		113	" HIGHER HOB AND BETTER UNIFORMITY
GUIDE COAT A	2° X 104° C	119	"
GUIDE COAT A	10° X 121° C	120	" SAME AS CONTROL
GUIDE COAT A	30° X 121° C	112	"
		112	

GUIDE COATING	DRY AMBIENT TEMP/BAKE	HEAD-ON-BRIGHTNESS	NOTE
CONTROL	(NO GUIDE COATING)	114	
GUIDE COAT B	30' X AMB. TEMP.	122	HIGHER HOB AND BETTER UNIFORMITY
GUIDE COAT B	2' X 104° C	124	"
GUIDE COAT B	10' X 121° C	113	SAME AS CONTROL
GUIDE COAT B	30' X 121° C	115	"

GUIDE COATING	DRY AMBIENT TEMP/BAKE	HEAD-ON-BRIGHTNESS	NOTE
CONTROL	(NO GUIDE COATING) 30' X AMB TEMP.	113	
GUIDE COAT C	2' X 104 C	123	HIGHER HOB AND BETTER UNIFORMITY
GUIDE COAT C	10' X 121 C	122	"
GUIDE COAT C	30' X 121 C	120	"
GUIDE COAT C		120	"

The above data shows that when no guide coating is used (control) head-on-brightness is noticeably lower than with a guide coating. Also, when the guide coats A and B are completely cured as occurs by baking for 10' at 121°C and for 30' at 121°C, head-on-brightness is substantially lower in comparison to only drying or partially curing the guide coat.

5 With guide coating C which is the acrylic latex without a crosslinking agent curing does not occur and the desirable improvement by use of this guide coat is noted at all baking temperatures.

Claims

10 1. A process for applying a multilayer finish to a substrate having a primer layer, the multi layer finish comprising a base coating composition layer and a layer of a clear coating composition adhering to the base coating; characterized by;

15 applying a layer of a guide coating composition comprising an aqueous carrier and a polymeric binder to the primer layer of the substrate and drying said composition without crosslinking or curing said guide coating composition, applying a base coating composition to the guide coating and a clear top coating composition to the base coating and baking at elevated temperatures to cure fully the resulting multilayer finish.

20 2. A process according to claim 1 in which the guide coating composition comprises a binder of a polymeric latex, and an aqueous carrier, and optionally a crosslinking agent.

25 3. A process according to claim 2 in which the guide coating composition comprises a binder of an acrylic polymer latex, an alkylated melamine formaldehyde crosslinking agent, metallic flake pigments and an aqueous carrier.

4. A process according to claim 3 in which the acrylic polymer of the latex contains hydroxyl and carboxyl groups.

5. A process according to claim 4 in which the acrylic polymer comprises an alkyl methacrylate, an alkyl acrylate, a hydroxy alkyl acrylate or methacrylate and an ethylenically unsaturated carboxylic acid.

6. A process according to claim 5 in which the acrylic polymer comprises methyl methacrylate, butyl acrylate, hydroxy ethyl acrylate and methacrylic acid.

30 7. A process according to claim 5 or 6 in which the acrylic polymer contains a difunctional acrylate or methacrylate.

8. A process according to claim 7 in which the acrylic polymer contains allyl methacrylate.

9. A process according to any one of claims 1 to 8 in which the guide coating composition is dried at about 20-185°C.

35 10. A process according to any one of claims 1 to 8 in which the guide coating composition and the base coating composition are curable at ambient temperatures.

11. A process according to any one of claims 1 to 10 in which the guide coating composition and the base coating composition are water-borne and the guide coating composition and/or the base coating composition incorporate metallic flake pigments.

40 12. A guide coating composition comprising 10-75% by weight of binder and 25-90% by weight of an aqueous carrier;

wherein the binder consists essentially of 60-95% by weight of a dispersed acrylic polymer consisting essentially of about 10-20% by weight based on the weight of the polymer of methyl methacrylate, 70-80% by weight, based on the weight of the polymer of butyl acrylate, 2-9% by weight, based on the weight of the polymer of hydroxy ethyl acrylate and 1-5% by weight based on the weight of the polymer of methacrylic acid;

45 5-40% by weight of an alkylated melamine formaldehyde resin and containing pigment in a pigment to binder ratio 0.5/100 - 300/100.

13. A primer coated substrate having a cured multilayer finish comprising a layer of the guide coating composition of claim 12 adhered to a primer coated substrate, a pigmented base coating adhered to the guide coating composition and a clear top layer adhered to the base coating.

50 14. A coated substrate according to claim 13 in which the guide coating has a thickness of about 0.003 to 0.025 mm (0.1 - 1.0 mils), the base coating has a thickness of about 0.005 to 0.038 mm (0.2 - 1.5 mils) and the clear top coating has a thickness of about 0.013 to 0.152 mm (0.5 - 6 mils).



EP 87 31 1191

DOCUMENTS CONSIDERED TO BE RELEVANT												
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.4)									
A	US-A-3 953 643 (CHEUNG et al.) * Column 2, line 34 - column 3, line 15; column 6, lines 51-62 * ---	1,2,4,5 ,6,11, 12	B 05 D 7/16 B 05 D 7/26									
D,A	US-A-3 639 147 (BENEFIEL et al.) * Column 13, lines 55-65; claim 1 * ---	1										
A	US-A-4 619 746 (DELANEY et al.) * Column 1, lines 42-57; column 2, lines 3-16 * ---	1,2										
A	FR-A-2 244 572 (I.C.I. LTD) * Claims 1,4 *	1										
A	US-A-4 303 581 (LEVINE et al.) * Abstract *	3										
E	US-A-4 731 290 (CHANG) * Claims *	1-14										
			TECHNICAL FIELDS SEARCHED (Int. Cl.4)									
			B 05 D									
<p>The present search report has been drawn up for all claims</p> <table border="1"> <tr> <td>Place of search THE HAGUE</td> <td>Date of completion of the search 21-07-1988</td> <td>Examiner MCCONNELL C.H.</td> </tr> <tr> <td colspan="2">CATEGORY OF CITED DOCUMENTS</td> <td>T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document</td> </tr> <tr> <td colspan="2">X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document</td> <td></td> </tr> </table>				Place of search THE HAGUE	Date of completion of the search 21-07-1988	Examiner MCCONNELL C.H.	CATEGORY OF CITED DOCUMENTS		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document	X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document		
Place of search THE HAGUE	Date of completion of the search 21-07-1988	Examiner MCCONNELL C.H.										
CATEGORY OF CITED DOCUMENTS		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document										
X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document												

